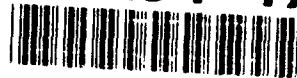


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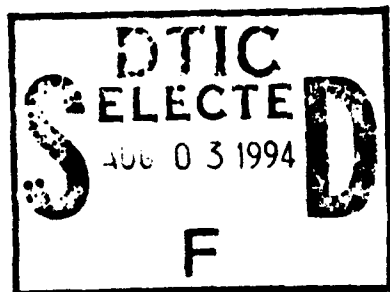
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26 June 1960

PRODUCTION AND THE PHYSICAL METALLURGY
OF PURE METALS -- PART IV

- USSR -

by A. I. Yevstyukhin



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PRODUCTION AND THE PHYSICAL METALLURGY
OF PURE METALS -- PART IV

INVESTIGATION OF THE PROCESS OF THERMAL REDUCTION
OF BeF_2 WITH MAGNESIUM

[This is a translation of an article written by A. I. Yevstyukhin in Metallurgiya i Metallovedeniye Chistykh Metallov (Production and the Physical Metallurgy of Pure Metals), No. 1, Moscow, 1959, pages 91-105.]

The problem of beryllium metallurgy has not as yet been solved. Beryllium is the subject of a great number of works [1/; however, little is known about it and it is not used on an industrial scale except in small quantities as an alloying component in a number of nonferrous alloys.

Intensive studies of beryllium are being made because of its happy combination of valuable structural properties: low specific weight, approximately one-third less than the specific weight of aluminum (1.84 and 2.70); relatively high melting point, approximately twice that of aluminum (1,287° and 660°C); high resistance to corrosion in air, approximately equal to the corrosion resistance of aluminum; and finally, beryllium has a very high elasticity modulus, approximately four times that of aluminum and 1.5 times higher than iron.

Semi-industrial production of beryllium was first organized in 1928, exactly 130 years after its discovery. Hopes for its use in airplane construction were not justified as the metal could not be obtained in a malleable state.

The method of preparing it--high temperature electrolysis of fused beryllium oxyfluoride ($3\text{BeO} \cdot 5 \text{BeF}_2$)--was difficult technically and toxic for the servicing personnel. Due to this fact, beryllium production by the above-mentioned method was suspended, although the investigation of beryllium metallurgy was continued without interruption in many countries of the world.

Interest in pure metallic beryllium arose again during the Second World War in connection with the work dedicated to atomic energy. It was found that along with valuable

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structural properties, the metal also has a number of valuable physical properties; it has high conductivity for x- and gamma rays (approximately 17 times higher than aluminum); it can be used as neutron source when irradiated by alpha rays; and of all the metals, it has the smallest cross section of the atom for capture of thermal neutrons (only 10.009 barns). Therefore, beryllium is a nearly ideal material for slowing thermal neutrons.

In connection with the atomic studies during the war and in the years after the war, the production of metallic beryllium by two methods was organized: 1) low temperature chloride electrolysis and 2) metallothermic reduction of beryllium fluoride by magnesium.

It was possible by both methods to prepare a metal of relatively high purity from metallic admixtures. But in both cases, beryllium was not sufficiently malleable. Simple items could be prepared by methods of powder metallurgy or by casting under vacuum, but it was impossible to manufacture such articles as, for instance, thin-walled tubes for nuclear reactors.

Although at present there are no exact data on the reasons for beryllium brittleness, the majority of researchers consider that beryllium brittleness can be ascribed to oxygen dissolved in the metal, which concentrates at the grain boundaries and renders the metal brittle. As oxygen is soluble in beryllium and forms solid solutions with it, it is possible that the brittleness of metal also depends on the oxygen present in the solid solution.

Pure beryllium uncontaminated by oxygen is, very probably, a ductile metal. In making such an assumption, reference is made to zirconium which, like beryllium, has a hexagonal lattice with very similar parameters and a low ratio of axes (for Zr, $c/a = 1.590$; for Be, $c/a = 1.585$; see Fig. 1 and the table following).

For a long time zirconium also was considered to be a brittle metal until a method of refining it was worked out permitting the preparation of a metal with an oxygen content of one hundredth or one thousandth part of one percent. However, beryllium with such an oxygen content has not as yet been prepared; samples of metallic beryllium of the highest purity contain from 0.1 to 0.5% oxygen. Therefore one can suppose that beryllium purified from oxygen (to the same degree as has been achieved for zirconium) also will be ductile. Refining by distillation under vacuum does not purify beryllium fully from oxygen, evidently because of the volatility of beryllium oxide. Other methods of beryllium refining have not as yet been devised, but research continues.

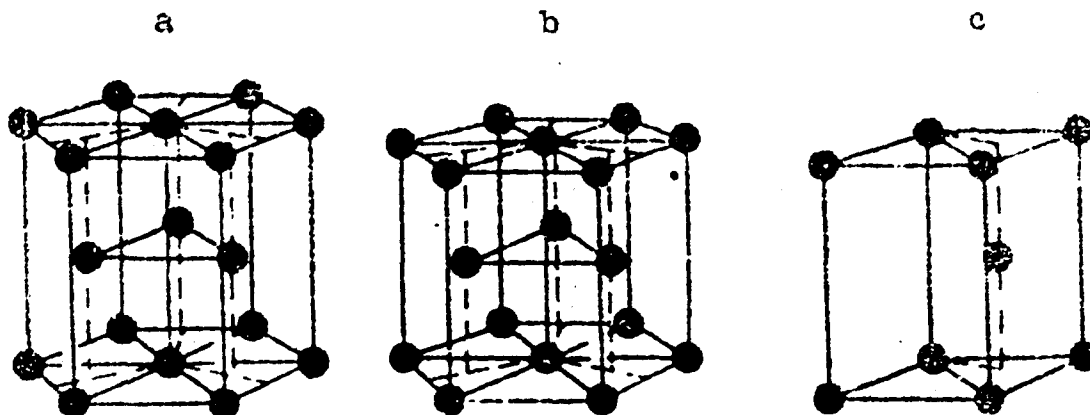


Fig. 1. Crystal lattices of zirconium and beryllium: a - hexagonal/zirconium lattice with close atom packing ($c/a = 1.590$); b - hexagonal/beryllium lattice with close atom packing ($c/a = 1.585$); c - elementary beryllium lattice cell.

For beryllium refining and its use in technical grades, a cheap initial metal is required. Until now beryllium has been one of the most expensive metals. It is about 500 times more expensive than aluminum and 6.5 and 17 times more expensive than zirconium and titanium respectively.

The high cost of beryllium was and still is one of the reasons preventing its wide use in industry.

From an economic comparison of existing methods for the production of beryllium, one can assert that less expensive metal can be prepared by the magnesium-thermal method, which presents a number of advantages over the method of fluoride electrolysis.

The advantages of the magnesium-thermal method can be briefly enumerated as follows:

1. The initial raw product for the magnesium-thermal method, BeF_2 , is prepared by a simpler and cheaper method than the BeCl_2 required for electrolysis.

2. Beryllium prepared by the magnesium-thermal method is in a solid piece, rather than in flakes, as in electrolysis; it is known that flake-shaped beryllium absorbs more gases and is oxidized to a greater extent than solid metal pieces with less developed surface area.

3. The rate of the magnesium-thermal process is higher than the rate of electrolysis and assures greater

Table

Constants of beryllium and zirconium lattices according to different researchers $/1/$; $/2/$

Beryllium			Temperature, °C	Modification	Zirconium		
Lattice Constants A°		c/a			Lattice Constants A°		c/a
a	c				a	c	
2,283	3,6100	—	20	α	3,230	5,140	1,59
2,283	3,6100	—	20-852		3,227	5,137	1,59
2,28105	3,57711	—	—		3,223	5,123	1,59
2,2685	3,5242	1,5847	20	β	3,229	5,141	1,59
2,2812	3,5773	1,5685	20		3,223	5,123	1,59
2,2608	3,5735	—	more than 865		3,16	—	—
2,2812	—	1,5682	more than 962		3,01	—	—
—	—	—	—		3,61	—	—
—	—	—	more than 865		3,12	—	—

productivity of an aggregate, and the whole method is more economical. According to published data, by the end of 1954 nearly all metallic beryllium produced in the U. S. A. was prepared by the magnesium-thermal method /3/.

However, at the present time, the magnesium-thermal method has a number of flaws, the first of which is inherent in the method itself and unavoidable. To prepare pure beryllium, pure initial components are required. Metallic magnesium must be distilled under vacuum (sometimes twice); beryllium fluoride must be carefully purified from admixtures by chemical analytical methods which are complicated and costly.

The second drawback is the low direct yield of beryllium due to the fact that approximately half of the BeF_2 charged is used as a flux-forming material, and is not reduced by magnesium. This drawback apparently could be eliminated if another flux-forming material having properties similar to those of BeF_2 were substituted for the latter. To do so, a detailed study of the mechanics of the process of reduction and the physical chemistry of formed fluxes must be carried out. However, studies in this area are absolutely inadequate.

THERMODYNAMICS AND CHEMISTRY OF BeF_2 REDUCTION BY MAGNESIUM

Metallothermal reduction of BeF_2 by magnesium is expressed in general way by the reaction



The heat effect Q_1 of this reaction is positive and equals the difference of between the BeF_2 and MgF_2 heats of formation. At room temperature, the heat effect of reaction (1) is equal to 36 kcal. However, it is known that it is not always possible to predict by the magnitude and sign of a heat effect at room temperature how a reaction will run, as the magnitude of the effect and its sign changes with increasing temperature.

Thermodynamic possibility of reducing BeF_2 is determined by the chemical affinity of the reducing metal for fluorine, which is measured by the magnitude of free energy of fluoride formation.

Metallic beryllium can be displaced from its fluoride in cases when the free energy of fluoride formation by the reducing metal is higher than that of BeF_2 . The magnitude of free energy of formation of a compound depends on the temperature of the reaction. This dependence at atmos-

pheric pressure for certain reactions taking place in thermal reduction of BeF_2 by magnesium has been calculated by the author and is represented graphically in Fig. 2.

From that figure it appears that the free energy of fluoride formation decreases with increasing temperature. The absolute quantity of free energy for BeF_2 in the whole temperature range is higher than for MgF_2 , CaF_2 and BaF_2 , but it is lower than for AlF_3 , MnF_2 , CrF_2 , FeF_2 , NiF_2 and CuF_2 .

From the above it follows that a metallothermal reduction of BeF_2 may be achieved with such metals as magnesium, calcium and barium but not with metals such as aluminum, manganese, chromium, iron, nickel and copper. Conversely, the latter metals accompanying BeF_2 as admixtures will be reduced by magnesium in the first place (as compared to BeF_2). Therefore, in the production of metallic beryllium, the initial fluoride must be free of these admixtures.

It is interesting to note that free energies of formation of fluoride compounds of rare earths have graphs considerably lower than BeF_2 and even MgF_2 . Thus, thermodynamically, rare-earth fluorides cannot be reduced by magnesium and must pass into the slag. As an example, Fig. 2 shows curves of free energy of formation for lanthanum and gadolinium fluorides (LaF_3 and GdF_3).

Although long known (see, for instances, references in work /4/) and apparently simple, the magnesium-thermal reduction of BeF_2 could not be carried out for a long time due to the high rate and exothermal character of the reaction. During reduction, the temperature in the reaction zone rose higher than boiling point of magnesium, magnesium and vapors burned and caused explosions. To avoid this, the reduction had to be carried out at a temperature lower than the boiling point of magnesium. In practice this could not be achieved because of the following circumstances.

At a stoichiometric ratio of BeF_2 and Mg , plus 10% excess of the latter (as is usually employed in metallothermal reactions) a hard crust of slag consisting of MgF_2 is formed in which melted BeF_2 particles are trapped, preventing magnesium from entering into the reaction. Only after raising the furnace temperature above the melting point of Be does the product of the reaction begin to soften and sag in the crucible. Unreacted Mg contacts superheated BeF_2 and reenters into a reaction which is accompanied by small explosions.

On the other hand, after a multitude of small particles of reduced Be are melted, they are also trapped in the crust of hard slag and form a slag-metal phase which is difficult to separate.

kJ/mol F_2

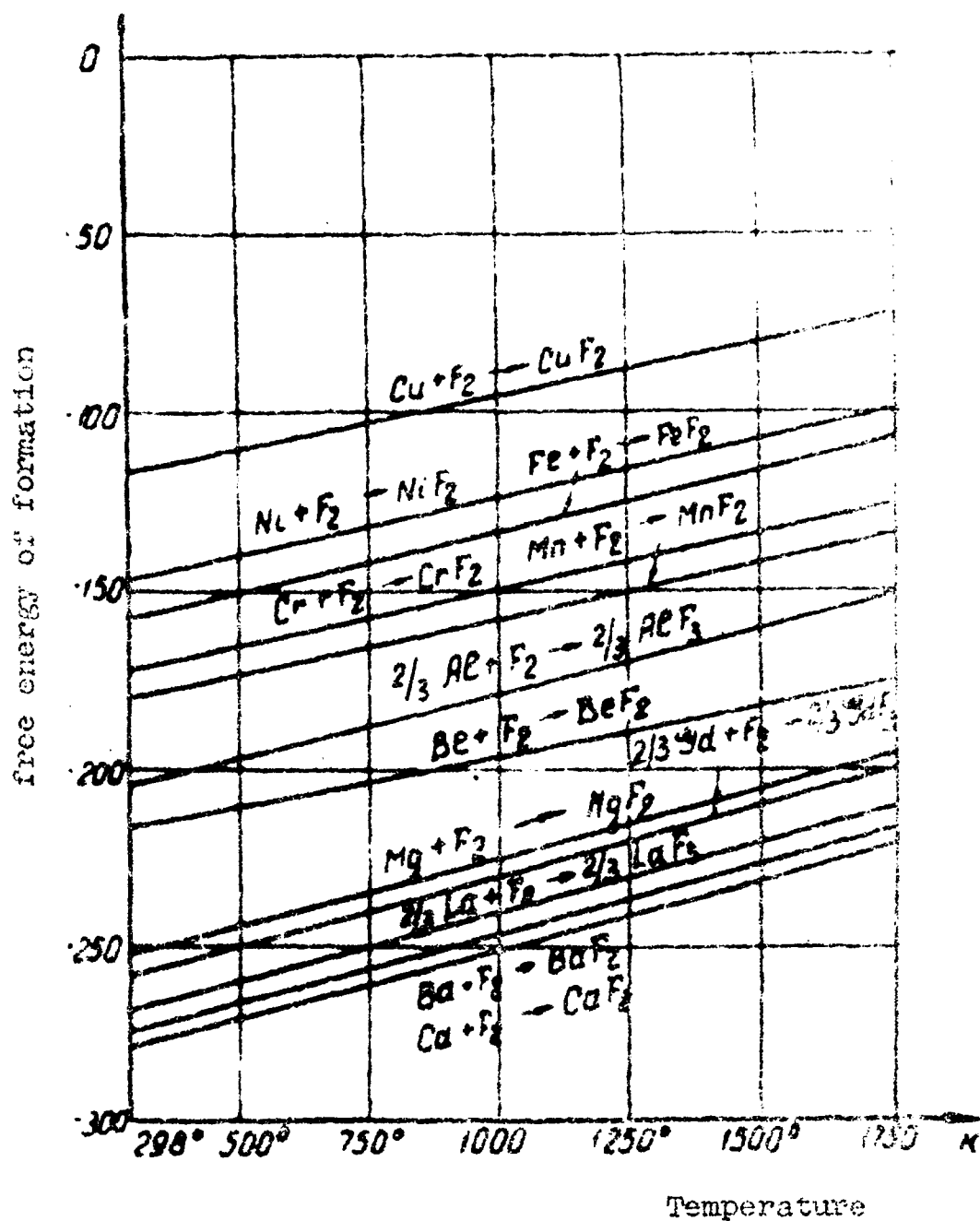


Fig. 2. Values of free energy of formation of BeF_2 as well as of other metal fluorides, versus temperature.

These difficulties were eliminated after the introduction into the charge (BeF_2 and Mg) of a flux lowering the melting point of slag being formed.

This creates favorable conditions for the agglomeration of melted beryllium particles rising to the surface of liquid slag. Melted beryllium "layer" after solidifying forms an ingot which can be extracted from liquid slag which has not yet solidified.

At the same time, the flux introduced into the charge removes excessive heat by its melting and thus lowers the temperature and the rate of the reduction, prevents explosions, and makes the reaction more feasible in practice. The change of phase composition of solid charge and of reaction products in the crucible can be followed.

Metallic magnesium melts at 651° and boils at 1126°C . BeF_2 , being a vitreous substance, has no definite melting point but softens and becomes liquid at 950° , whereas its boiling point according to data in the literature and to our observations, lies around $1,300^\circ\text{C}$.

The specific weight of solid BeF_2 at 20° is 1.986. The specific weight of BeF_2 in molten state was not determined, but by analogy with other fluorides one can expect that it will be only very slightly lower. The specific weight of magnesium at its melting point is 1.584. Thus, when melted, magnesium will float on the top of liquid BeF_2 with which it does not mix.

The initial temperature of BeF_2 reduction by magnesium according to our observations and determinations is within the range of 950 - $1,000^\circ\text{C}$.

From the above it follows that reduction of BeF_2 by magnesium apparently takes place at the interface of these two liquid phases.

The reaction products Be and MgF_2 are solids at the temperature of reduction. Melting and boiling points of metallic Be at atmospheric pressure are, respectively, 1287 and 2507°C , while those of MgF_2 are 1396 and 2239°C .

The specific weight of Be is 1.85, whereas the specific weight of MgF_2 is 2.9. Magnesium fluoride formed as a result of reduction at the interface of two unmixable liquid phases (BeF_2/Mg) must sink in melted BeF_2 and entrain with it particles of solid metallic Be .

The remaining Be particles which have a specific weight less than BeF_2 and higher than melted Mg float at the interface of the liquid phases. Melting of MgF_2 and simultaneous formation of liquid slag with an excess of BeF_2 takes place at the end of reduction when the furnace temperature is raised above the melting point of Be to agglomerate the latter at the surface of the slag. A schemat-

ic diagram of the phase situation in the crucible before, at the end of, and after the reduction reaction is shown in Fig. 3.

When the temperature of the furnace rises above the boiling point of magnesium, the vapors of this metal flare up. Magnesium excess on the slag surface is burned out without reducing the BeF_2 tied up in liquid slag and situated outside the sphere of action of metallic magnesium vapors.

Slag Composition and the Binary System $\text{BeF}_2\text{-MgF}_2$

In the present article the author presents the result of his study of the content of slag which is formed during BeF_2 reduction by magnesium when BeF_2 is used as flux-forming component.

In this case, the slag consists of two components, MgF_2 and BeF_2 . To rationally select slag components for this process one must know the state diagram of the system BeF_2 and MgF_2 .

There are no data in literature concerning studies of the above-mentioned system except a publication in the Journal of Abstracts /5/ which states that this system was studied by M. Venturello of the Academy of Science of Torino (Italy) /6/. To his regret the author could not acquaint himself with this work, and from a short abstract one could not draw conclusions as to its results; therefore it was decided to study the system in the laboratory.

During the investigation, difficulties arose due to the amorphous state of BeF_2 , its volatility at high temperature, the decomposing action of humidity and oxygen, and an interaction of fluorine compound vapors with the apparatus. These difficulties were successfully overcome with the help of methods applied to the analysis of similar fused systems. The method consisted in a thermal analysis of alloys with differential recording of thermograms in an atmosphere of argon followed by radiographic, metallographic and chemical analyses.

The BeF_2 used for the investigation was prepared from chemically pure BeO , HF and NH_4F with subsequent decomposition of ammonium fluoroberyllate. Melted BeF_2 was an amorphous, vitreous and only slightly hygroscopic substance.

MgF_2 was prepared from chemically pure MgO and HF . It was a definitely crystalline subject for a substance with a tetragonal structural lattice.

Inasmuch as one of the components (BeF_2) was a non-crystalline substance that hardened as a supercooled liquid

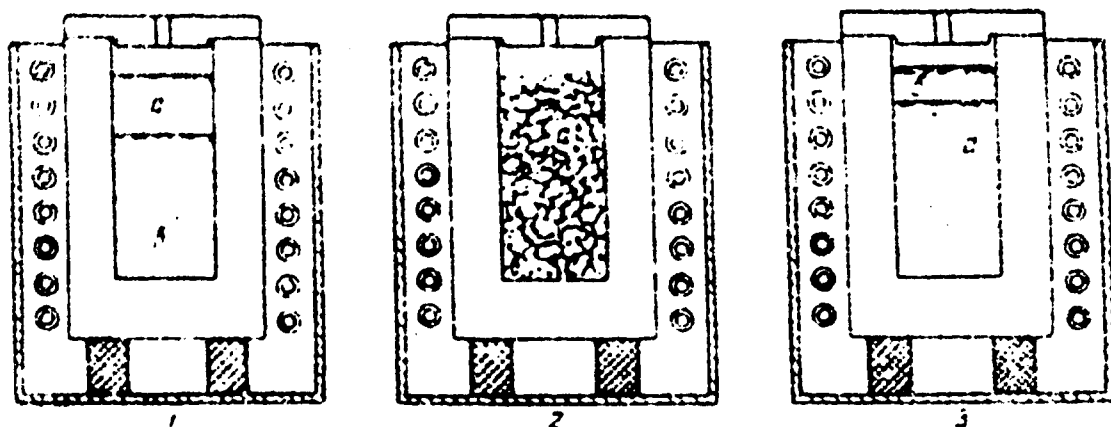


Fig. 3. Schematic diagram of phase distribution in the crucible.

1. Before the reduction: a) molten Mg (spec. wt. 1.584);
b) molten BeF_2 (spec. wt. 1.986).
2. After completed reduction: a) solid MgF_2 (spec. wt. 2.9); b) solid Be (spec. wt. 1.85); c) molten BeF_2 (spec. wt. 1.985).
3. After phase separation by heating: a) $\text{BF}_2 / \text{MgF}_2$ liquid slag; b) molten Be.

and the other component (MgF_2) was crystalline, data obtained from thermal analysis were insufficient for plotting a diagram. Therefore in this case radiological and microstructural analyses were of great importance and especially useful.

Chemical analysis established the composition of alloys after melting of the charge, as the composition of the charges of these alloys changed but slightly due to BeF_2 evaporation. Figure 4 shows a diagram plotted on the basis of thermal radiographic and microstructural analyses of BeF_2 - MgF_2 systems. As it appears from the figure, the state diagram of the BeF_2 - MgF_2 system clearly expresses only the liquidus line which stretches over the whole range of concentrations of initial components. The solidus line was not ascertained by thermal analysis over the whole range of concentration, owing to the small effect of MgF_2 dry-stallization; thus, this diagram is substantially a

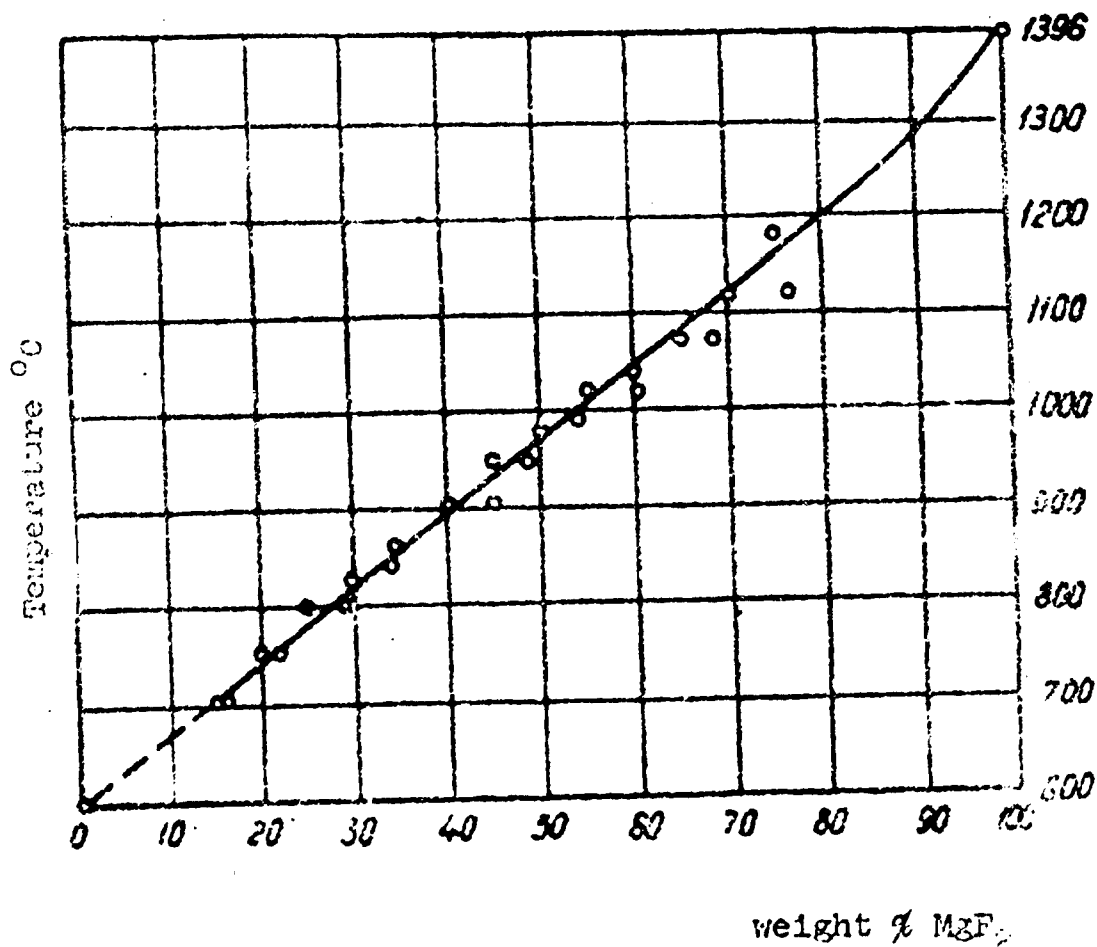


Fig. 4. State diagram of system $\text{BeF}_2\text{-MgF}_2$.

diagram of MgF_2 solubility in BeF_2 . This is clearly shown by slides of the alloy (Fig. 5), as well as by the schematic diagram of line intensity in radiograms (Fig. 6).

At low weight concentration of MgF_2 (in the range from 0 to 15%) we have not succeeded in ascertaining the position of solubility line and therefore it is shown by dots. Points from 0 to 15% MgF_2 are not shown at all on the diagram, as neither radiographic nor microstructural analyses have proved the presence of MgF_2 , while thermograms of alloys with this composition do not show any thermal effects and are completely analogous to thermograms of pure BeF_2 .

It is evident that the effects of crystallization of small quantities of MgF_2 in a viscous BeF_2 melt

are so small that they are outside the sensitivity range of the thermocouple.

However, at high MgF_2 concentration the thermograms of the BeF_2 - MgF_2 alloy system indicate the presence of only one heat effect corresponding to MgF_2 crystallization over the whole range of concentrations. This heat effect increases with increasing concentration of MgF_2 . The absence of heat effects corresponding to crystallization of the second component indicates that BeF_2 remains a non-crystalline compound over the whole range of concentrations, and that the crystallization of MgF_2 does not influence its solidification. It is evident that MgF_2 crystallizing out does not provide crystallization centers for BeF_2 , which solidifies as a vitreous substance (quite analogously to its action when in pure form). This conclusion is fully confirmed by the data of radiographic and microstructural analyses of alloys investigated.

The measurement of radiogram lines of the alloys discloses that the crystal lattice of MgF_2 does not change its parameters and is not distorted. However, the radii of magnesium and beryllium atoms differ considerably (the radius of the Be atom being 1.42 times smaller than that of Mg). This fact indicates that solid BeF_2 is practically absolutely insoluble in solid MgF_2 . The solubility of solid MgF_2 in solid BeF_2 also could not be established, as the maximum blackening of radiographic lines corresponding to BeF_2 is considerably washed out.

Mutual solubility of the BeF_2 - MgF_2 system of components in solid state could not be ascertained by thermal analysis, which confirms the correctness of the conclusion concerning the character of the diagram. When fused, BeF_2 and MgF_2 have a mutual unlimited solubility, but after solidifying and cooling to room temperature, the solution changes into a mechanical mixture from which it is easy to wash out with water soluble BeF_2 . Analytical methods for determination of the composition of alloys after melting was based on this property.

In plotting the diagram of BeF_2 - MgF_2 the volatility factor of BeF_2 at high temperature was taken into account by a chemical analysis of alloy composition after melting and cooling. The line of solubility on the diagram is plotted according to the points of the chemical analysis. In general, it must be noted that according to our data, the volatility of BeF_2 is low in absolute terms and rises only slightly with an increase in temperature. Therefore, in plotting the diagram it could practically be ignored.

The plotted diagram of the unbalanced system BeF_2 - MgF_2 does not raise any doubts and is, apparently, very

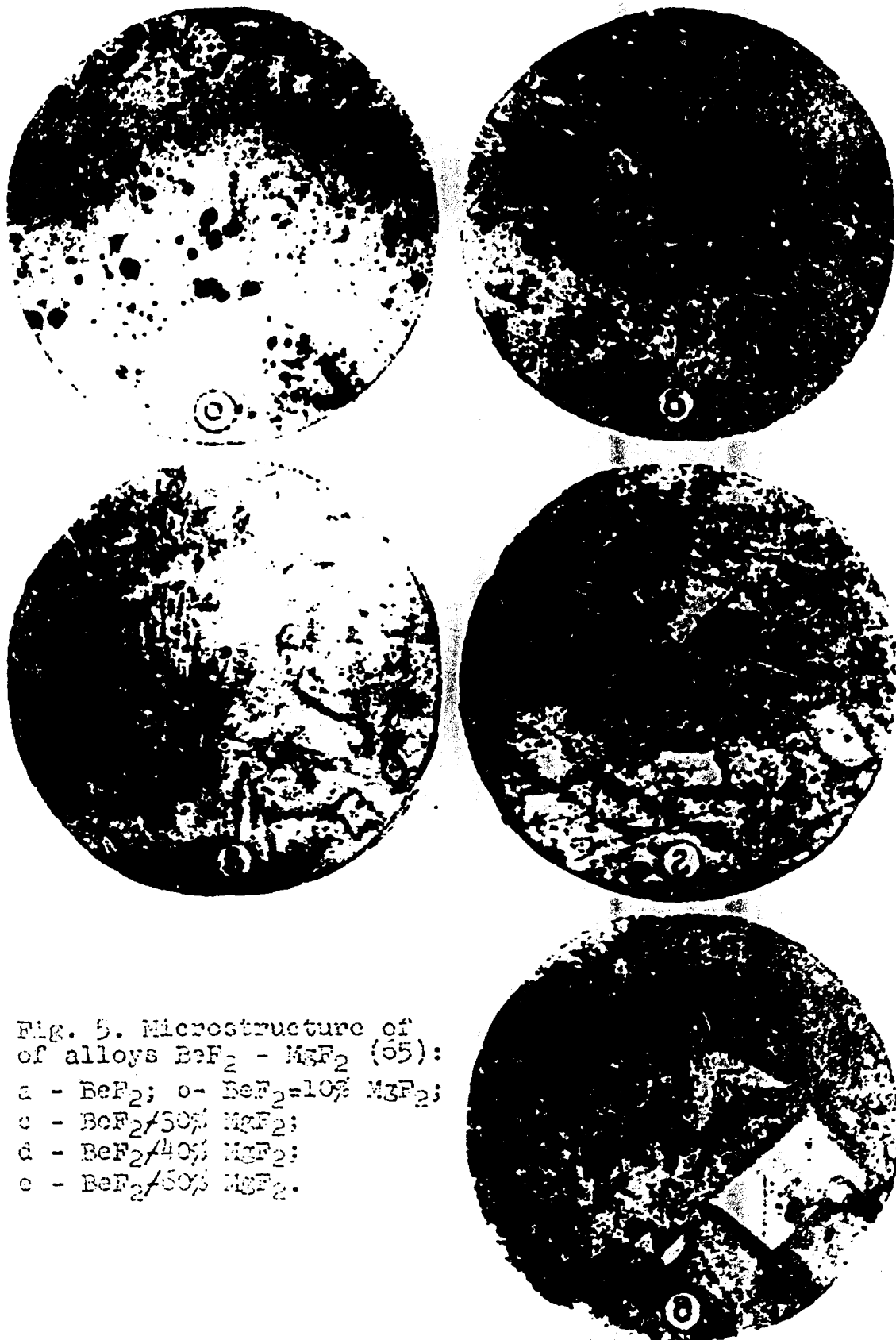


Fig. 5. Microstructure of
of alloys BeF₂ - MgF₂ (55):
a - BeF₂; b - BeF₂=10% MgF₂;
c - BeF₂/30% MgF₂;
d - BeF₂/40% MgF₂;
e - BeF₂/50% MgF₂.

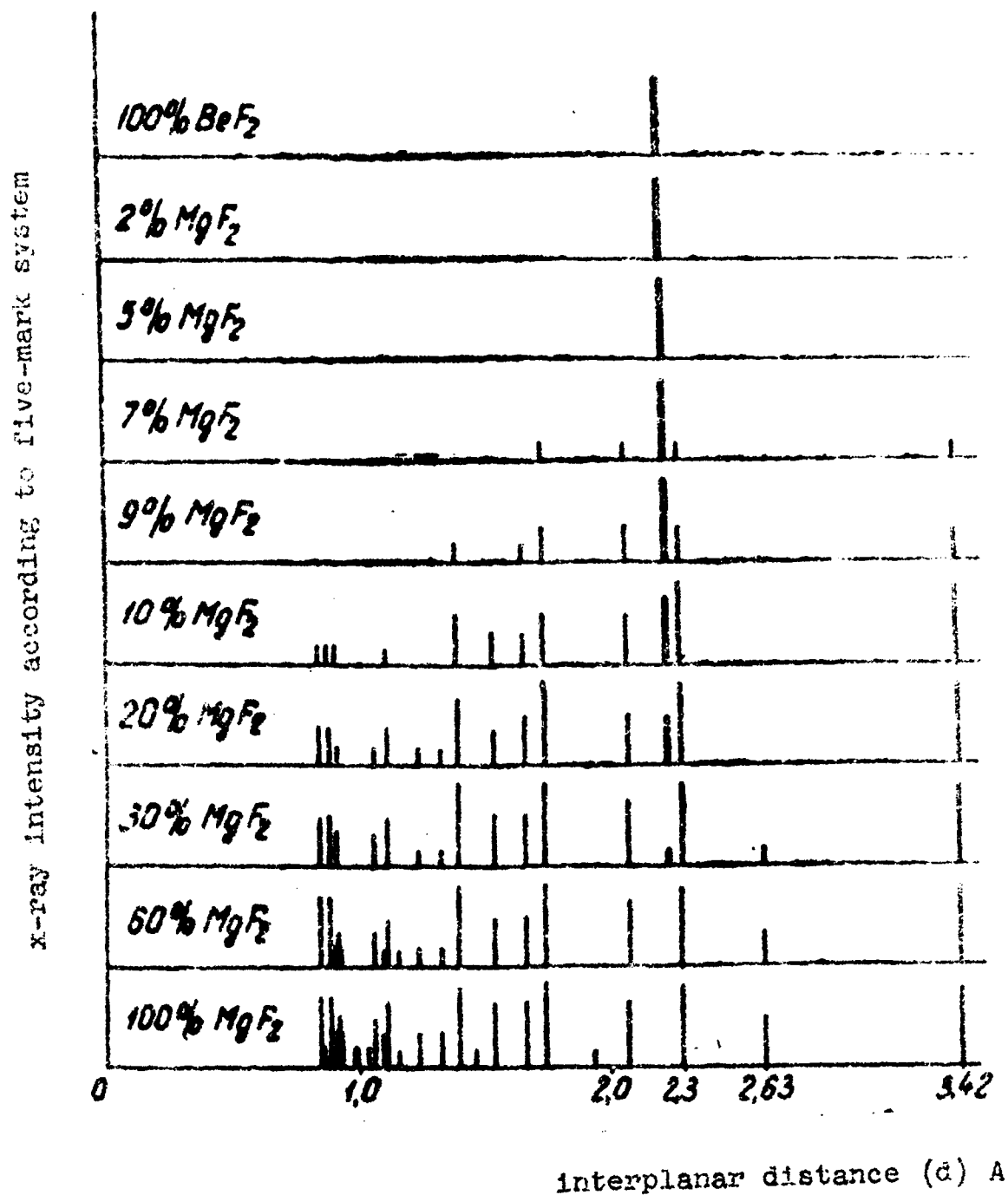


Fig. 6. Schematic diagram of line intensity in radiograms of BeF_2 - MgF_2 alloys.

close to reality. Considering that in the given system one of the components (BeF_2) remains noncrystalline over the whole temperature range and solidifies as a vitreous substance, the viscosity or fluidity of the BeF_2 - MgF_2 system (and consequently the slag viscosity) depends on temperature and must change along with the change in content of the high-melting compound. A study of viscosity as a function of composition and temperature is of great importance.

Flux-forming Additions and Their Influence on the Chemistry and Kinetics of the Process

As mentioned before, the efficiency of a magnesium-thermal method for preparing beryllium can be doubled if BeF_2 flux is replaced by some equivalent-quality but less costly material.

The flux-forming additive must meet the following strict requirements:

1. It must form a low melting system with the MgF_2 formed;
2. It must form a slag which, without wetting melted beryllium, would induce the agglomeration of its small particles into drops on the surface;
3. It must not be reduced by magnesium, and the metal entering into it should not alloy with beryllium;
4. It must be a relatively inexpensive material;
5. It must have a sufficiently high degree of purity.

The author undertook a systematic review and experimental verification of flux-forming materials for the process under study. It was found that only the fluorides of certain alkali-earth metals meet the above requirements. These compounds together with BeF_2 form stable complexes which are not reduced by magnesium. Fluorides of metals of other groups of the periodic system are reduced by magnesium and cannot be used in this case.

Attempts by the author to use chlorides of alkali-earth metals as flux, particularly MgCl_2 , were unsuccessful. Slags containing chlorides wet the melted beryllium and hinder its agglomeration at the surface.

In one of the methods offered by Kowecki /7/ it is recommended that CaF_2 be added as the flux-forming addition to the charge. Reduction is carried out in a fused mixture containing approximately equimolar quantities of BeF_2 and CaF_2 over a temperature range of 700 to 1000°C, i. e., lower than boiling point of magnesium.

The quantity of magnesium for reduction is taken 10% higher than required according to the stoichiometric ratio, and the beryllium yield is nearly theoretical.

Due to the fact that calcium forms alloys with beryllium, it is partly reduced and contaminates the metal obtained by this method. The refining of metallic beryllium from calcium admixtures is very difficult, even by distillation under vacuum.

The author proposes employing BaF_2 as a flux-forming additive, considering that barium does not alloy with beryllium and does not contaminate it as does BeF_2 when reduced by magnesium.

Tests were carried out for reducing BeF_2 by magnesium with BaF_2 . The beryllium yield was near to theoretical and the reaction ran smoothly without explosions at a temperature of 900 to 1000°C, i. e., lower than boiling point of magnesium.

Conclusions

1. A brief characterization of the state of beryllium metallurgy at the present moment is given.
2. A thermodynamic calculation for reducing BeF_2 by magnesium was made, and the mechanics of the reduction were discussed in connection with phase changes in the initial charge.
3. An experimental study of the state diagram in binary system $\text{BeF}_2\text{-MgF}_2$ was carried out for the purpose of selecting a rational composition for the slag to be used in magnesium-thermal reduction of BeF_2 which uses an excess of BeF_2 as a flux-forming component.
4. The influence of the nature of the slag on the mechanics and kinetics of BeF_2 reduction by magnesium was discussed. Conditions were reviewed which are imperative for a satisfactory flux-forming addition. A systematic review and experimental verification of flux-forming additions in magnesium-thermal reduction of BeF_2 was carried out.

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